National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1649a

Urban Dust

Standard Reference Material (SRM) 1649a is an atmospheric particulate material collected in an urban area and is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and total carbon in atmospheric particulate material and similar matrices. Reference and information values are also provided for selected polychlorinated dibenzo-p-dioxin and dibenzofuran congeners, inorganic constituents, total extractable material, mutagenic activity, particle-size characteristics, and chemical and isotopic (^{14}C) carbon composition. All of the constituents for which certified, reference, and information values are provided in SRM 1649a are naturally present in the particulate material. SRM 1649a is the same particulate material that was issued previously in 1982 as SRM 1649 [1]; this material has been rebottled and reanalyzed to provide updated certified values as well as certified, reference, and information values for additional constituents. A unit of SRM 1649a consists of a bottle containing 2.5 g of particulate material.

Certified Concentration Values: Certified values for the concentrations, expressed as mass fractions, for 22 PAHs, 35 PCB congeners (some in combination), and 8 chlorinated pesticides are provided in Tables 1, 2, and 3, and for total carbon in Table 12. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained from two or more chemically independent analytical techniques performed at NIST.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 22 additional PAHs in Tables 4 and 5 and for one additional chlorinated pesticide in Table 6. Reference values are provided in Table 7 for the seventeen 2,3,7,8-substituted polychlorinated dibenzo-p-dioxin and dibenzofuran congeners and total, tetra-, penta-, hexa-, and hepta-substituted congeners of polychlorinated dibenzo-p-dioxin and dibenzofuran. Reference values for 32 selected inorganic constituents are provided in Table 8. Reference values for mutagenic activity are provided in Table 9. Reference values for particle-size characteristics and total extractable mass are provided in Tables 10 and 11, respectively. Reference values for chemical carbon and isotopic carbon (^{14}C) composition are summarized in Tables 12 and 13, respectively. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as a note in Tables 4 through 13.

Information Concentration Values: Information concentration values, expressed as mass fractions, for selected components of the chemical and isotopic carbon composition are provided in Tables 12 and 13. An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed. Explanations in support of the information values are provided in the footnotes for Tables 12 and 13.

Expiration of Certification: The certification of this SRM lot is valid until **30 June 2007**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The support aspects involved in the preparation, recertification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald and T.E. Gills.

Willie E. May, Chief
Analytical Chemistry Division

Nancy M. Trahey, Chief
Standard Reference Materials Program

Coordination of the technical measurements leading to the updated certification was under the direction of S.A. Wise of the NIST Analytical Chemistry Division. Coordination of the measurements and evaluation of the data for chemical and isotopic carbon composition were performed by L.A. Currie and G.A. Klouda of the NIST Surface and Microanalysis Science Division.

Analytical measurements for the certification of SRM 1649a were performed by B.A. Benner, Jr., A. Deissler, R.R. Greenberg, M.J. Hays, B.J. Porter, D.L. Poster, L.C. Sander, M.M. Schantz, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division. Analytical measurements for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among 13 laboratories (see Appendix A) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, Ontario, Canada). Contributors to the analytical measurements for isotopic and particulate carbon composition are listed in Appendix B. The results were derived primarily from an interlaboratory comparison study coordinated by L.A. Currie, with assistance from G.A. Klouda and J.D. Kessler of the NIST Surface and Microanalysis Science Division. The particle-size distribution data were provided by Honeywell, Inc.,[1] Clearwater, FL.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.S. Levenson, S.B. Schiller, and M.G. Vangel of the NIST Statistical Engineering Division.

The collection and preparation of the material for SRM 1649a were supported in part by the U.S. Environmental Protection Agency, Office of Health Research.

NOTICE AND WARNING TO USERS

Storage: SRM 1649a is provided in amber glass bottles and should be stored away from direct sunlight at room temperature or below.

Handling: This material is naturally occurring urban atmospheric particulate matter and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Instructions for Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1649a are generally reported on a dry mass basis. The SRM, as received, contains approximately 1.2 % moisture. A separate subsample of the SRM should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry mass basis.

PREPARATION AND ANALYSIS

Sample Collection and Preparation: This SRM was prepared from atmospheric particulate material collected in the Washington DC area in 1976-1977 using a baghouse specially designed for the purpose. The particulate material was collected over a period in excess of 12 months, and therefore represents a time-integrated sample. While the sample is not intended to be representative of the area in which it was collected, it should generally typify atmospheric particulate matter obtained from an urban area. The particulate material was removed from the baghouse filter bags by a specially designed vacuum cleaner and combined into a single lot. This lot was passed through a 125 μm (120 mesh) sieve to remove bag fibers and other extraneous materials. The sieved material was then thoroughly mixed in a V-blender and bottled.

Conversion to Dry Mass Basis: The results for the constituents in SRM 1649a are reported on a dry mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1649a was determined by measuring the mass loss after freeze drying subsamples of 1.6 g to 2.5 g for five days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1649a at the time of the certification analyses was 1.23 % ± 0.07 % at the 95 % confidence level.

Polycyclic Aromatic Hydrocarbons (Tables 1, 4, and 5): The general approach used for the determination of PAHs in SRM 1649a was similar to that reported for the recent certification of several environmental matrix SRMs [2-5]. This approach consisted of Soxhlet extraction using dichloromethane or a hexane/acetone mixture followed

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[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

by analysis of the extract using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on a 5 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase, and (4) GC/MS analysis of the PAH fraction on a smectic liquid crystalline stationary phase. These procedures are described in detail for SRM 1649a in Reference [6] and are described briefly below. Additional results for selected PAHs were obtained by pressurized fluid extraction (PFE) followed by GC/MS analysis on a 50 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase.

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Subsamples of 3 g from six bottles were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated and then processed through an aminopropylsilane solid phase extraction (SPE) cartridge to obtain the total PAH fraction. Approximately one third of this fraction was analyzed as the total PAH fraction; the second portion of the extract was then fractionated on a semi-preparative aminopropylsilane column to isolate isomeric PAH fractions as described previously [7-10]. The total PAH fraction and the isomeric PAH fractions were analyzed using both 3 µm and 5 µm particle size polymeric octadecylsilane (C_{18}) columns (4.6 mm i.d. × 15 cm, 3 µm particle size, ChromSpher PAH, Chrompack, Middelburg, The Netherlands and 4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [2,8,9].

Five sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), and GC/MS (sm), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of 1 g from 10 bottles were Soxhlet extracted for 20 h with dichloromethane. The concentrated extract was passed through an aminopropylsilane SPE cartridge and eluted with 2 % dichloromethane in hexane. The PAH fraction was then isolated from the extract using normal-phase LC [7-10] on a semi-preparative aminopropylsilane column. The PAH fraction was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses consisted of subsamples from six bottles analyzed as a second sample set using the same preparation and analysis procedures as described for GC/MS (I). Two additional sets of GC/MS results for a limited number of PAHs, designated as GC/MS (III) and GC/MS (IV), were obtained using PFE followed by GC/MS. For the GC/MS (III) analyses subsamples of 0.4 g to 1 g from six bottles were extracted with dichloromethane using PFE, as described in Reference [11], the extracts were processed as described above for GC/MS (I), followed by GC/MS analysis on a 50 % phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (IV) analyses, two subsamples of 1 g each were extracted with each of three different solvents (dichloromethane, acetonitrile, and 50 % hexane/50 % acetone mixture) using PFE [11], the extracts were processed and analyzed by GC/MS on the 5 % phenyl-substituted methylpolysiloxane stationary phase described above for GC/MS (I). The GC/MS (sm) results were obtained by analyzing selected sample extracts from the GC/MS (I) set on a 0.2 mm i.d. × 25 m (0.15 µm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT). The liquid crystalline phase provides significantly different selectivity for the separation of PAH isomers when compared with the 5 % phenyl-substituted methylpolysiloxane phase [2]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the particulate matter prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1649a was assessed by analyzing duplicate samples of 1 g from 10 randomly selected bottles. Samples were extracted, processed, and analyzed as described above for the GC/MS (I). No statistically significant differences between bottles were observed for the PAHs at the 1 g sample size. Analyses of subsamples of 1 mg to 400 mg show no significant differences in the PAH concentrations, and a sample size of approximately 450 mg will contribute less than 1 % error due to sample homogeneity for the PAHs for which certified values are provided.

PCBs and Chlorinated Pesticides (Tables 2, 3, and 6): SRM 1649a was analyzed for selected PCB congeners and chlorinated pesticides using gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity and using GC/MS. This same approach has been used previously for the certification of PCBs and chlorinated pesticides in environmental matrix SRMs [3,5,12,13]. For the GC-ECD analyses, subsamples of approximately 1 g from each of six bottles were Soxhlet extracted for 18 h using dichloromethane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and, (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W

Scientific, Folsom, CA) and a 0.32 mm × 100 m fused silica capillary column with a dimethylpolysiloxane phase containing 50 % (mole fraction) C-18 dimethylpolysiloxane (0.1 µm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands).

A second set of samples was also analyzed by GC-ECD; however, these samples were extracted using PFE with dichloromethane as described in Reference [11]. Subsamples of 1 g to 2 g were extracted using approximately 15 mL of dichloromethane. The extract was processed and analyzed by GC-ECD on the 5 % phenyl-substituted methylpolysiloxane phase as described above.

For the GC/MS analyses, subsamples of 0.5 g to 1.5 g each from six randomly selected bottles were mixed with 50 g of precleaned sodium sulfate and Soxhlet extracted for 18 h using 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated to 1 mL and then placed on a precleaned silica SPE column and eluted with 15 mL of 10 % dichloromethane in hexane. The concentrated eluent was analyzed by GC/MS on a 5% phenyl-substituted methylpolysiloxane phase as described above. For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the air particulate extract (PCB 103 and PCB 198 [14,15]), and 4,4'-DDT-d_8 were added to the air particulate material prior to extraction for use as internal standards for quantification purposes. The analyses of SRM 1649a for the determination of PCBs and pesticides is described in detail in Reference [16].

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans (Table 7): Value assignment of the concentrations of the seventeen 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta- substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1649a by 13 laboratories that participated in an interlaboratory comparison study (see Appendix A). Each laboratory analyzed three subsamples (typically 0.5 g) of SRM 1649a using their routine analytical procedures and high resolution gas chromatography with high resolution mass spectrometry detection (GC-HRMS). The analytical procedures used by all of the laboratories included spiking with ^{13}C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and, finally, analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % cyanopropylphenyl-substituted methylpolysiloxane (DB-225, J&W Scientific, Folsom, CA) capillary column.

Inorganic Constituents (Table 8): The majority of the inorganic constituents were determined using instrumental neutron activation analysis (INAA). For INAA, duplicate 100 mg subsamples from six bottles of SRM 1649a were analyzed. Selected trace elements (copper, lead, magnesium, manganese, nickel, and vanadium) were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES). For the ICP-AES analyses, duplicate 250 mg subsamples from four bottles of SRM 1649a were analyzed. For the determination of chlorine and sulfur, 250 mg subsamples from six bottles of SRM 1649a were analyzed using high pressure oxygen bomb combustion followed by ion chromatography.

Mutagenic Activity (Table 9): The reference values for the mutagenic activity of a dichloromethane extract of SRM 1649a were determined as part of an international collaborative study in 1989 sponsored by the International Programme on Chemical Safety (IPCS). The IPCS is jointly sponsored by the World Health Organization (WHO), the United Nations Environmental Programme (UNEP), and the International Labor Organization (ILO). The program was initiated, supported, and technically coordinated by the U.S. Environmental Protection Agency's Office of Health Research. Twenty laboratories from North America, Europe, and Japan participated in the study for which a complete summary is available [17,18]. As part of the protocol, each laboratory used dichloromethane to extract the organic material from SRM 1649a. Half of the laboratories used Soxhlet extraction and the other half used ultrasonic extraction procedures. The extracted material was analyzed using the Salmonella/mammalian microsomal plate-incorporation assay using strains TA98 and TA100 [19]. The mean dichloromethane extractable mass determined in the IPCS collaborative study was 5.0 % ± 0.4 %, and the extract was found to be mutagenic in both strains with and without activation in all 20 laboratories.

Two types of suggested Bioassay Reference Values are provided in Table 9. The first value is the best estimate, from the data available, of the mutagenic activity for a dichloromethane extract of SRM 1649a using the protocol specified for the IPCS collaborative study. For the reference values to apply, the sample should be Soxhlet or ultrasonically extracted with dichloromethane. The dichloromethane extract should be evaporated to near dryness and solvent exchanged to dimethylsulfoxide. The bioassay procedure should follow the *Salmonella typhimurium* plate-incorporation protocol as described in Reference [19] and adhere to the guidelines provided in Reference [20]. Minimal media plates should be made of Difco agar and should contain 30 mL ± 1 mL of base layer agar. The

exogenous activation system (S9) should be an Aroclor-1254 induced rat liver homogenate as described in Reference [19]. Duplicate plates should be used for each of 3 to 5 dose levels.

The uncertainty in the mutagenic activity, expressed as 95 % confidence limits about the mean potency value, takes into account both between and within laboratory sources of variation. While these confidence limits represent the uncertainty for the best estimate of the mutagenic activity of SRM 1649a, they do not reflect the variation in the values reported by individual participating laboratories. They should also not be taken to represent the range of mutagenic activity values from other laboratories using the protocol described in Reference [19] with some additional constraints [21]. Prediction intervals [22] are provided to characterize differences in the mutagenic activity reported by the 20 laboratories that participated in the IPCS interlaboratory study and to establish a target range for other laboratories that analyze SRM 1649a using the modified Marion and Ames protocol. Additionally, for the investigator values to be assessed using the tolerance limits given, data should be treated using the same or very similar statistical methods as those used in this study [23,24][2].

An "80 % Prediction Interval" is a range within which 80 % of the mutagenic activity values reported in the interlaboratory study are expected to reside. These limits may be used by all laboratories using the IPCS Salmonella bioassay protocol to determine if their findings are consistent with those reported by the 20 laboratories that participated in the IPCS collaborative study. Although these laboratories may not be representative of all laboratories that conduct the Salmonella bioassay, the tolerance limits given do provide a range of values that all laboratories following the IPCS protocol should strive to obtain. The first set of tolerance limits given are for laboratories that use the same number of replicate extractions and bioassays as were performed in the IPCS collaborative study. The second set of tolerance limits, which are slightly wider, apply to the case where only a single extraction and bioassay is performed.

Particle-Size Information (Table 10): Dry particle-size distribution measurements for SRM 1649a were obtained as part of a collaborative effort with the Honeywell Particle and Components Measurements Laboratory, Clearwater, FL. A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1649a. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle-size as a volume distribution is derived via computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle-size distribution are calculated. The system has a working range from 0.7 µm to 700 µm.

Extractable Mass (Table 11): For the determination of extractable mass, six samples of approximately 15 g of SRM 1649a were Soxhlet extracted with 250 mL of 50 % hexane/50 % acetone (volume/volume) for 20 h. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in mass before and after extraction was determined.

Isotopic and Particulate Carbon Composition (Tables 12 and 13): Laboratories and analysts contributing to the isotopic and particulate carbon composition results are listed in Appendix B. Data for this section were compiled by L.A. Currie and G.A. Klouda of the NIST Surface and Microanalysis Science Division.

Nomenclature [26]: Nomenclature for "total" and "elemental" fractions of particulate carbon tends to be ambiguous and varied across disciplines. As used here, "total carbon (TC)" refers to the total, non-carbonate fraction of particulate carbon; as such, it includes what are commonly called "organic" and "elemental" carbon fractions. The meaning of TC is identical to what is frequently called "total organic carbon (TOC)" in some areas of geoscience, a term used to highlight the removal of inorganic (carbonate) carbon.

Problems with metrology and nomenclature are even more serious for the Refractory Pyrogenic Carbon (RPC) phase labeled EC here. As a result, only information values can be given for this constituent at this time. Among the terms having wide usage are: char, black carbon, elemental carbon, soot carbon, refractory carbon, and graphitic carbon [27]. In some cases, these terms have specialized meanings that are method-specific or material/structure-specific; in other cases, they are used interchangeably. For a discussion and recommendations concerning the RPC nomenclature problem, see Reference [26].

Thermal-Optical-FID Method (SLI, NIST): In a completely oxygen-free helium atmosphere, the sample (~1 mg) was

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[2]A computer program developed by the U.S. Environmental Protection Agency to run under MS-DOS entitled *GeneTox Manager* contains the statistical analysis software developed by several research groups for the Salmonella assay including the program described by Krewski et al. [23,24]. The *GeneTox Manager* has been described [25] and is available from the U.S. EPA through a written request to: Dr. L.D. Claxton, Environmental Carcinogenesis Division, MD-68, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

heated in four increasing steps (from approximately 500 °C to 900 °C) to remove all organic carbon on the filter. Organic compounds that are pyrolytically converted to elemental carbon were continuously monitored by measuring the transmission of helium-neon laser light through the filter. As organic compounds are volatilized, they are immediately oxidized to carbon dioxide (CO_2) using a plug of manganese dioxide (MnO_2) at 700 °C, reduced to methane over nickel on firebrick in the presence of hydrogen (H_2), and measured using a flame ionization detector (FID). After cooling the sample to approximately 525 °C, a 2 % or 5 % oxygen(O_2)/helium(He) mixture was introduced and the temperature increased to approximately 850 °C. Based on the FID response and laser-transmission data, the amounts of organic, elemental, and pyrolytic carbon are then calculated for the sample [28].

Combustion-GC-TCD (NIST):   Samples (0.3 mg to 9 mg) were weighed into aluminum boats, combusted to carbon dioxide at 900 °C in an atmosphere of oxygen, purified by gas chromatography (GC), quantified with GC using a thermal conductivity detector (TCD) [29].

Combustion-NDIR (NIST):   The weighed sample was placed in a ceramic crucible which was then purged with oxygen while inductively heating the crucible. The carbon monoxide (CO) and carbon dioxide produced were measured using a non-dispersive infrared (NDIR) detector [30].

Combustion-CHN (SU, URI):   Samples were placed in silver boats and fully oxidized and quantified by flash combustion/gas chromatographic analysis using a commercial CHN-analyzer. This process served also as the final carbon quantification step for the "Gustafsson procedure" to determine elemental ("soot") carbon [SC]. For SC assay, the final CHN combustion step was preceded by thermal oxidation for 24 h at 375 °C in the presence of excess oxygen and treatment with hydrochloric acid (HCl) for carbonate removal if necessary [31].

H_2O Extraction, Combustion-Manometry (NIST) [29]:   Subsamples of SRM 1649a were placed on prefired quartz filters. Three 200 mL portions of prefiltered distilled water were passed through the sample and filter. The filters were dried at 60 °C for 2 h before closed tube combustion with copper oxide (CuO). The resulting carbon dioxide was distilled and quantified by manometry and then ^{14}C analysis was performed at UA [32].

H_3PO_4 Acidification-Manometry (NIST):   A weighed sample (280 mg) and 2 mL of 100 % phosphoric acid were placed in opposite branches of an inverted Y-shaped tube. The stem of the Y-tube was adapted to a vacuum manifold. The system was evacuated, and the acid was frozen by applying dry ice/isopropanol slurry. Vacuum was applied to the frozen acid for a short time, then the acid was warmed to room temperature. The system was closed to vacuum, then the acid was poured onto the sample and the mixture heated with a beaker of boiling water for 2 to 3 h. A sample bulb immersed in liquid nitrogen was used to collect the evolved carbon dioxide, which was then measured using manometry as described above [29].

Soxhlet Extraction (EPA) [30]:   Samples were Soxhlet extracted for 24 h with dichloromethane. The solvent was removed by rotary evaporation and the extract was reconstituted to 10 mL with dichloromethane. Gravimetric determinations were then made by removing a 200 µL aliquot of the extract solution, evaporating the solvent, and weighing the residue to constant weight. A 1 mL aliquot of the extract solution was evaporated to dryness in a quartz tube. Copper oxide and silver wire were then added to the tube. The tube was attached to a vacuum line, evacuated to 5 Pa, sealed, and the contents combusted to carbon dioxide at 900 °C. The sample carbon dioxide was then reduced to a graphite-iron bead [33] for ^{14}C AMS measurements at UA [32].

Combustion-Manometry (NIST) [34]:   Samples were combusted to carbon dioxide in a quartz furnace filled with 101 kPa oxygen. Downstream of the combustion furnace is a series of three furnaces: (1) platinum gauze at 900 °C, (2) copper oxide at 800 °C, and (3) silver wool at 400 °C to assure complete combustion and to purify the carbon dioxide of sulfur and halogen containing impurities. The sample gas stream is then reduced to less than 13 kPa to prevent the condensation of liquid oxygen by controlling the gas flow through the system using a throttle valve and a vacuum pump. Before the vacuum pump, the sample carbon dioxide is cryogenically trapped at liquid nitrogen temperature (-196 °C) in a series of spiral glass traps. The resulting carbon dioxide is cryogenically separated from other gaseous combustion products by distillation from -78 °C and quantified using manometry in a calibrated volume. Low level ^{14}C decay counting was performed on the carbon dioxide using a miniature gas proportional counter at NIST [35,36].

H_3PO_4-Combustion-Manometry (UCI) [37]:   A subsample of SRM 1649a, silver foil (prefired at 550 °C), and copper oxide wire (prefired at 850 °C) were added to a quartz tube. Approximately 5 mL of 3 % phosphoric acid (H_3PO_4) as added to the tube to remove any inorganic carbon. The quartz tube was then attached to a vacuum line, evacuated to a pressure of less than 5 Pa, sealed, and combusted to carbon dioxide at 850 °C for 4 h. The carbon dioxide was reduced to graphite over cobalt catalyst at 850 °C in the presence of hydrogen. Accelerator mass spectrometry ^{14}C measurements were performed at LLNL [38].

Combustion-GC (NIST) [39]:   Samples were placed in tin boats and fully oxidized and quantified by flash combustion/gas chromatographic analysis using a commercial CHN-analyzer, by essentially the same procedure used for TC quantification by Combustion-GC-TCD. The purified carbon dioxide was then trapped at -196 °C and transferred to UA for preparation of a graphite target [40] and ^{14}C AMS [32].

Soxhlet Extraction/LC Isolation of Aromatic Fraction (NIST):   Samples were Soxhlet extracted for 24 h with dichloromethane. The extract was concentrated to a small volume under a stream of nitrogen. The concentrated extract was placed on a silica SPE cartridge and eluted with 10 % dichloromethane in pentane. The aromatic fraction was isolated using NPLC on an aminopropylsilane column as described above. This fraction was concentrated and a 1 mL aliquot transferred to a quartz tube and evaporated to dryness. Copper oxide was added to the tube which was then attached to a vacuum line, evacuated, sealed, and the contents combusted to carbon dioxide. Low-level ^{14}C decay counting was performed on the sample carbon dioxide using a miniature gas proportional counter at NIST [35,36].

Soxhlet Extraction/LC/PCGC (NIST, WHOI):   Extraction followed by LC was used for isolating Polar, Aromatic, and Aliphatic fractions for ^{14}C analysis. The aromatic fraction was specially purified using silica gel flash chromatography and LC PAH ring size fractionation [7], prior to separation and collection of individual PAH using an automated preparative capillary GC (PCGC) system described in Reference [41].   The individual PAH fractions were then subjected to closed-tube combustion and ^{14}C determined by AMS [26,42-44].

[14]C Methods for Elemental Carbon [26]
Thermal oxidation/residue (WHOI): [14]C is measured in the residual carbon after thermal oxidation at 375 °C for 24 h (to remove labile organic carbon) and acidification (to remove inorganic carbonates). The residual carbon is placed in a quartz tube containing copper oxide and elemental silver and combusted at 850 °C for 5 h. The ^{14}C content of the resulting carbon dioxide was measured by accelerator mass spectrometry [44].
Chemical oxidation, dichromate/residue (UCI):   Wet oxidization with $0.1M$ $Cr_2O_7^=$ in $2M$ H_2SO_4 at 50 °C was performed for periods up to 406 h. The ^{14}C AMS result is given for the residual carbon for the longest (406 h) reaction period. Because of the gentler oxidation treatment (chemical vs. thermal), the ^{14}C must reflect somewhat less refractory components of the "black carbon spectrum" [45].
Thermal kinetic oxidation/intercept (NIST):   EC is defined as the refractory (intercept) component that survives isothermal oxidation at 560 °C in a stream of helium (5 % oxygen). The intercept is estimated by fitting a five parameter model (2 exponentials + intercept) to the residual carbon rate function. Intercept ^{14}C is estimated as the end point of a series of three intermediate samples taken for ^{14}C AMS [46].

Other Thermal EC Methods (LBNL, LSCE, DRI, TUW):   Four additional thermal and thermal-optical methods were employed by laboratories contributing to the EC/TC results in Table 12.   Two- to multi-stage thermal steps were employed, with the earlier stages including lower temperature oxidation (air, oxygen) or volatilization (helium), and the later stages including high temperature oxidation (air, oxygen, helium-oxygen), and in some cases laser transmission or reflectance to correct for charring [47-50]. Full details are given in the review of the international comparison [26].

Table 1. Certified Concentrations for Selected PAHs in SRM 1649a[a,b]

	Mass Fractions (in mg/kg)		
Phenanthrene[c,d,e,f]	4.14	±	0.37
Anthracene[c,d,e,f]	0.432	±	0.082
Fluoranthene[c,d,e,f]	6.45	±	0.18
Pyrene[c,d,e,f]	5.29	±	0.25
Benz[a]anthracene[c,d,h]	2.208	±	0.073
Chrysene[e,h]	3.049	±	0.060[i]
Triphenylene[e,h]	1.357	±	0.054
Benzo[b]fluoranthene[e,f,g]	6.45	±	0.64
Benzo[k]fluoranthene[c,d,e,f,g]	1.913	±	0.031
Benzo[a]fluoranthene[c,d,e]	0.409	±	0.035
Benzo[e]pyrene[c,d,e]	3.09	±	0.19
Benzo[a]pyrene[c,d,e,f]	2.509	±	0.087
Perylene[c,d,e,f]	0.646	±	0.075
Anthanthrene[c,d,e,h]	0.450	±	0.067
Benzo[ghi]perylene[c,d,g,h,j]	4.01	±	0.91
Indeno[1,2,3-cd]pyrene[c,d,g,h,j]	3.18	±	0.72
Dibenz[a,j]anthracene[c,d,g,h]	0.310	±	0.034
Dibenz[a,c]anthracene[e,g,h]	0.200	±	0.025
Dibenz[a,h]anthracene[e,g,h]	0.288	±	0.023
Pentaphene[c,d,g,h]	0.151	±	0.035
Benzo[b]chrysene[c,d,e,g,h]	0.315	±	0.013
Picene[c,d,e,g,h]	0.426	±	0.022

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.

[b] Each certified value is the equally-weighted mean of the means from two or more independent analytical methods. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values for the certified value, within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[c] GC/MS (I) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

[d] GC/MS (II) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

[e] GC/MS (sm) analysis using a smectic liquid crystalline phase after Soxhlet extraction with dichloromethane.

[f] LC-FL analysis of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[g] GC/MS (III) analysis on 50 % phenyl-substituted methylpolysiloxane after PFE with dichloromethane.

[h] LC-FL analysis of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[i] The uncertainty interval for chrysene was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

[j] GC/MS (IV) analysis on 5 % phenyl-substituted methylpolysiloxane phase after PFE with three different solvents (dichloromethane, acetonitrile, and 50 % hexane/50 % acetone mixture).

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 1649a[b,c]

			Mass Fractions (in µg/kg)		
PCB	8	(2,4'-Dichlorobiphenyl)	12.28	±	0.29
PCB	18	(2,2',5-Trichlorobiphenyl)	20.44	±	0.84
PCB	28	(2,4,4'-Trichlorobiphenyl)	18.5	±	1.2
PCB	31	(2,4',5-Trichlorobiphenyl)	17.3	±	1.4
PCB	44	(2,2'3,5'-Tetrachlorobiphenyl)	15.4	±	1.6
PCB	49	(2,2'4,5'-Tetrachlorobiphenyl)	12.2	±	1.5
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)	24.65	±	0.97
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)[d]	65	±	12
	95	(2,2',3,5',6-Pentachlorobiphenyl)[d]			
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)	10.65	±	0.62
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)[e]	51.6	±	4.2
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl)	9.58	±	0.69
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)	52.9	±	1.0
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)	8.63	±	0.80
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)	26.6	±	1.6
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)	25.7	±	1.5
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	6.35	±	0.69
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	69.7	±	7.5
	163	(2,3,3',4'5,6-Hexachlorobiphenyl)			
	164	(2,3,3',4',5',6-Hexachlorobiphenyl)			
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl)	75.7	±	1.3
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)	34.3	±	3.9
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	82.5	±	8.0
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)	16.25	±	0.77
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	30.8	±	2.2
	190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)			
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	78.7	±	8.2
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)	20.34	±	0.95
PCB	187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	40.1	±	2.5
	159	(2,3,3',4,5,5'-Hexachlorobiphenyl)			
	182	(2,2',3',4,4',5,6'-Heptachlorobiphenyl)			
PCB	194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	28.9	±	3.6
PCB	195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl)	9.63	±	0.37
PCB	206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	20.6	±	4.6
PCB	209	Decachlorobiphenyl	8.04	±	0.77

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [14] and later revised by Schulte and Malisch [15] to conform with IUPAC rules; for the specific congeners identified in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the conditions used, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.

[c] Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel[52]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random uncertainty due to the drying study. The expanded uncertainty defines a range of values, within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[d] Concentration for PCB 95 and PCB 66 together was determined using GC-ECD on the 5 % phenyl-substituted methylpolysiloxane phase.

[e] Concentration for PCB 95 was determined using GC-ECD on the 50 % C-18 dimethylpolysiloxane phase and GC/MS on the 5 % phenyl-substituted methylpolysiloxane phase.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1649a[a,b]

	Mass Fractions (in µg/kg)		
Hexachlorobenzene	16.3	±	1.8
trans-Chlordane (γ-Chlordane)	40.3	±	2.8
cis-Chlordane (α-Chlordane)	34.88	±	0.42
trans-Nonachlor	27.6	±	1.6
2,4'-DDE	5.79	±	0.85
4,4'-DDE	40.4	±	1.7
4,4'-DDD	34.01	±	0.48
4,4'-DDT	212	±	15

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel[52]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random uncertainty due to the drying study. The expanded uncertainty defines a range of values, within which the true value is believed to lie, at a level of confidence of approximately 95 %.

Table 4. Reference Concentrations for Selected PAHs in SRM 1649a
as Determined by GC/MS[a,b]

These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. The associated uncertainties may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

	Mass Fractions (in mg/kg)		
Fluorene[c]	0.23	±	0.05
Dibenzothiophene[d]	0.18	±	0.01
1-Methylphenanthrene[c,d,e]	0.37	±	0.04
2-Methylphenanthrene[c,d,e]	0.73	±	0.12
3-Methylphenanthrene[c,d,e]	0.50	±	0.05
4-Methylphenanthrene and 9-Methylphenanthrene[c,d,e]	0.34	±	0.01
4H-Cyclopenta[*def*]phenanthrene[c,d,e]	0.32	±	0.06
Benzo[*c*]phenanthrene[d,e]	0.46	±	0.03
Benzo[*ghi*]fluoranthene[d]	0.88	±	0.02
Benzo[*j*]fluoranthene[d,f]	1.5	±	0.4
Indeno[1,2,3-*cd*]fluoranthene[e]	0.23	±	0.01
Benzo[*c*]chrysene[d]	0.080	±	0.004

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] The reference value for each analyte is the equally weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.
[c] GC/MS (I) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.
[d] GC/MS (sm) analysis using a smectic liquid crystalline phase after Soxhlet extraction with dichloromethane.
[e] GC/MS (II) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.
[f] GC/MS (III) analysis on 50 % phenyl-substituted methylpolysiloxane after PFE with dichloromethane.

Table 5. Reference Concentrations for Selected PAHs in SRM 1649a
as Determined by LC[a,b,c]

These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedure used, the reference values should be useful for comparison with results obtained using similar procedures.

	Mass Fractions (in μg/kg)		
Dibenzo[a,e]pyrene	630	±	80
Dibenzo[a,h]pyrene	53	±	2
Dibenzo[a,i]pyrene	130	±	10
Dibenzo[b,k]fluoranthene	800	±	100
Naphtho[2,3-a]pyrene	57	±	5
Naphtho[2,3-e]pyrene	240	±	30
Naphtho[2,3-b]fluoranthene	230	±	20
Naphtho1,2-k]fluoranthene	550	±	60
Naphtho[2,3-k]fluoranthene	57	±	3

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] Expanded uncertainties are sample standard deviations of the mean concentrations.
[c] Concentrations reported [110] were determined using LC-FL analysis of isomeric PAH fractions; duplicate analyses of two sample extracts.

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1649a[a]

The concentration is provided as a reference value because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

	Mass Fractions (in μg/kg)		
Heptachlor[b,c]	18.9	±	0.5[d]

[a] Concentration reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] GC-ECD analysis on the 50 % C-18 dimethylpolysiloxane phase.
[c] GC-ECD analysis on the 5 % phenyl-substituted methylpolysiloxane phase.
[d] The reference value is the equally-weighted mean of the means from two analytical methods. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

Table 7. Reference Concentrations for Selected Dibenzo-*p*-dioxin
and Dibenzofuran Congeners in SRM 1649a

These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

	Mass Fraction in µg/kg (Dry mass basis)[a,b]		
2,3,7,8-Tetrachlorodibenzo-*p*-dioxin	0.011	±	0.004[c]
1,2,3,7,8-Pentachlorodibenzo-*p*-dioxin	0.091	±	0.012
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	0.26	±	0.02
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin	0.68	±	0.05
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin	0.64	±	0.11
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	18.8	±	1.5
Octachlorodibenzo-*p*-dioxin	201	±	20
2,3,7,8-Tetrachlorodibenzofuran[d]	0.068	±	0.15[e]
1,2,3,7,8-Pentachlorodibenzofuran	0.090	±	0.010
2,3,4,7,8-Pentachlorodibenzofuran	0.28	±	0.03
1,2,3,4,7,8-Hexachlorodibenzofuran	0.87	±	0.26
1,2,3,6,7,8-Hexachlorodibenzofuran	0.43	±	0.06
2,3,4,6,7,8-Hexachlorodibenzofuran	0.69	±	0.03[f]
1,2,3,7,8,9-Hexachlorodibenzofuran	0.066	±	0.029[f]
1,2,3,4,6,7,8-Heptachlorodibenzofuran	3.8	±	0.2[f]
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.46	±	0.07[f]
Octachlorodibenzofuran	6.8	±	0.8[f]
Total Toxic Equivalents (TEQ)[g]	0.86	±	0.06
Total Tetrachlorodibenzo-*p*-dioxins	0.16	±	0.08[f]
Total Pentachlorodibenzo-*p*-dioxins	0.88	±	0.16
Total Hexachlorodibenzo-*p*-dioxins	6.0	±	0.5
Total Heptachlorodibenzo-*p*-dioxins	36.4	±	3.4
Total Tetrachlorodibenzofurans	0.52	±	0.13
Total Pentachlorodibenzofurans	1.6	±	0.2
Total Hexachlorodibenzofurans	5.0	±	0.6
Total Heptachlorodibenzofurans	9.8	±	0.8
Total Dibenzo-*p*-dioxins[h]	244	±	22
Total Dibenzofurans[h]	23.9	±	1.9

[a] Each reference value is the mean of the results from up to 13 laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO Guide [51] and k is the coverage factor. The value u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the reference value. Here u_c is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (12 unless noted otherwise). The coverage factor, k, is the value from a Student's *t*-distribution corresponding to a 95 % level of confidence.

[b] Concentrations reported on dry mass basis; material as received contains approximately 1.3 % moisture.

[c] Degrees of freedom = 9 for this compound.

[d] Confirmation results using a 50 % cyanopropylphenylpolysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl-polysiloxane phase columns.

[e] Degrees of freedom = 7 for this compound.

[f] Degrees of freedom = 11 for this compound.

[g] TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended in Reference [53]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

[h] Total of tetra- through octa- chlorinated congeners.

Table 8. Reference Concentrations for Selected Elements in SRM 1649a

These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique or only a limited number of analyses were performed; therefore, unrecognized bias may exist for some analytes in this matrix.

	Mass Fractions (in %)[a,b]		
Bromine[c]	0.119	±	0.001
Chlorine[d]	0.28	±	0.01
Iron[c,e]	2.98	±	0.07
Lead[e]	1.24	±	0.04
Magnesium[e]	0.92	±	0.03
Sulfur[d]	3.27	±	0.09
Zinc[c,e]	0.168	±	0.004

	Mass Fractions (in mg/kg)[a]		
Antimony[c]	29.9	±	0.7
Arsenic[c]	67	±	2
Barium[c]	569	±	21
Cadmium[c,e]	(22)[f]		
Cerium[c]	52	±	4
Cesium[c]	2.84	±	0.07
Chromium[c]	211	±	6
Cobalt[c]	16.4	±	0.4
Copper[e]	223	±	7
Europium[c]	0.87	±	0.07
Hafnium[c]	4.4	±	0.1
Lanthanum[c]	33	±	3
Manganese[e]	237	±	8
Molybdenum[c]	13.5	±	0.9
Nickel[e]	166	±	7
Rubidium[c]	48	±	3
Samarium[c]	4.7	±	0.4
Scandium[c]	8.7	±	0.2
Selenium[c]	25.6	±	0.7
Silver[c]	3.5	±	0.2
Thorium[c]	6.6	±	0.2
Tin[c]	56	±	13
Tungsten[c]	3.8	±	0.3
Uranium[c]	2.65	±	0.08
Vanadium[e]	345	±	13

[a] Concentration is reported on an as received basis; material as received contains approximately 1.2 % moisture.

[b] Each reference value is the mean of means of measurements made by instrumental neutron activation analysis (INAA) on duplicate subsamples from six bottles or measurements made by inductively coupled plasma atomic emission spectrometry (ICP-AES) on duplicate subsamples from four bottles, or the combination of results from both analytical techniques. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random uncertainty, as well as a Type B uncertainty of 1 % to 3 %. The expanded uncertainty defines a range of values with which the true value is believed to lie, at a confidence of approximately 95 %.

[c] Determined using INAA.

[d] Determined using high pressure oxygen bomb combustion and ion chromatography.

[e] Determined using ICP-AES.

[f] Cadmium value is the mean of the results from INAA (18.3 ± 1.1) mg/kg and ICP-AES (26.5 ± 1.0) mg/kg and is provided as an information value only because of the disagreement of the results from the two analytical techniques.

Table 9. Reference Values for Ames Bioassay Mutagenic Activity of SRM 1649a[a]

These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

| Strain/Activation | Mutagenic Activity[b] | 95 % Confidence Limits[c] | 80 % Prediction Intervals | |
			Multiple Extraction/ Bioassay[d]	Single Extraction/ Bioassay[e]
TA100, +S9	102 rev/mg	66 - 158	30 - 351	29 - 365
TA100, -S9	103 rev/mg	73 - 146	39 - 275	36 - 295
TA98, +S9	214 rev/mg	153 - 299	83 - 555	80 - 570
TA98, -S9	237 rev/mg	186 - 301	119 - 471	115 - 488

[a] Results summarized in Reference [21]. Reference values refer to the mutagenic activity of the dichloromethane extract of SRM 1649a per unit mass of the particulate material extracted. Doses for the IPCS collaborative study were based on the following mg equivalents of SRM 1649a particles: TA100, +/-S9 (0.25, 0.5, 1.0, 1.5, 2.0); TA98, +S9 (1.0, 2.0, 4.0, 6.0, 8.0); and TA98, -S9 (1.25, 2.5, 5.0, 7.5, 10.0). Total extractable mass for SRM 1649a with dichloromethane from the IPCS collaborative study was 5.0 % $\pm$ 0.4 % (mass fraction) as compared with 4.60 % $\pm$ 0.36 % with hexane/acetone in Table 10.

[b] Geometric mean of all replicate mutagenicity potency values reported by participating laboratories after deleting outlying observations. Results reported as revertants per mg of SRM 1649a.

[c] Calculated on a logarithmic scale taking into account both inter- and intra-laboratory variation, excluding outliers, and then re-expressed in the original scale by taking antilogs.

[d] Prediction intervals for mutagenic activity determined in a single laboratory using the same number of replicate extracts/bioassays as in the IPCS collaborative study.

[e] Prediction intervals for mutagenic activity determined in a single laboratory using only one replicate extraction/bioassay.

Table 10. Reference Values for Particle Size Characteristics for SRM 1649a

These results are provided as reference values because the results are method specific as defined by the procedure described in the Preparation and Analysis section. Although bias has not been evaluated for the procedure used, the reference values should be useful for comparison with results obtained using similar procedures.

Particle Measurement	Value[a]		
Mean diameter (volume distribution, MV, μm)[b]	34.6	$\pm$	0.4
Mean diameter (area distribution, μm)[c]	12.9	$\pm$	0.3
Mean diameter (number distribution, μm)[d]	1.50	$\pm$	0.09
Surface Area (m^2/cm^3)[e]	0.47	$\pm$	0.01

The following data show the percent of the volume that is smaller than the indicated size:

Percentile	Particle Diameter (μm)[a]		
95	100	$\pm$	3
90	73	$\pm$	1
80	49.9	$\pm$	0.2
70	38.3	$\pm$	0.1
60	30.5	$\pm$	0.2
50[f]	24.4	$\pm$	0.2
40	19.3	$\pm$	0.2
30	15.2	$\pm$	0.2
20	11.3	$\pm$	0.2
10	6.7	$\pm$	0.2

[a] The reference value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value, within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

[c] The mean diameter of the area distribution, calculated from the volume distribution with less influence by the presence of coarse particles than MV.

[d] The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

[e] Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

[f] Median diameter (50 % of the volume is less than 24.4 μm).

Table 11. Reference Value for Percent Extractable Mass in SRM 1649a

This result is provided as a reference value because results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedure used, the reference value should be useful for comparison with results obtained using similar procedures.

	Mass Fraction (in %)
Extractable Mass	4.6 $\pm$ 0.4[a,b,c]

[a] Concentration is reported on an as received basis; material as received contains approximately 1.2 % moisture.

[b] Extractable mass value was determined from Soxhlet extraction using 50 % hexane/50 % acetone (volume/volume).

[c] The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value, within which the true value is believed to lie, at a level of confidence of approximately 95 %.

Table 12. Values for Particulate Carbon Composition of SRM 1649a

This table includes certified (in bold), reference, and information values for chemical carbon composition of SRM 1649a. The certified value for *Total Carbon* (TC) is based on the agreement of results obtained from two or more independent analytical techniques. The reference value for *Extractable Mass* is method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedure used, the reference value should be useful for comparison with results obtained using similar procedures. Results for *Elemental*, *Insoluble*, and *Carbonate Carbon*, which currently do not satisfy the criteria for reference values, are provided as information values.

Chemical Component	Mass Fractions (in g/g)[a]		
Total Carbon (TC)[b]	**0.1768**	**±**	**0.0019**
Insoluble Carbon[c,d]	0.152		(*n* = 2)
Carbonate Carbon[d,e]	0.00118		(*n* = 2)
Extractable Mass[f,g]	0.044	± 0.006	(*n* = 3)

Elemental carbon, fraction of total (EC/TC)[h]	Median	Inter-quartile range	
Cluster-1	0.075	0.071, 0.078	(*n* = 4)
Cluster-2	0.28	0.27, 0.29	(*n* = 4)
Cluster-3	0.46	0.44, 0.50	(*n* = 5)
Complete range:		0.069, 0.520	(*n* = 19)

a Concentration is reported on an as received basis; material as received contains approximately 1.2 % moisture. For the identification and codes of laboratories contributing to the data, and literature citations for the reference methods, please consult the Preparation and Analysis section and Appendix B.

b The certified value for TC is the weighted mean of the means from two or more independent analytical methods. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method. The expanded uncertainty defines a range of values for the certified value which is expected to cover the true value with a level of confidence of approximately 95 %. Methods contributing to the certified value are Thermal-Optical-FID (SLI, *n* = 3; NIST, *n* = 2), Combustion-GC-TCD (NIST, *n* = 39), Combustion-NDIR (NIST, *n* = 15), Combustion-Coulometry (LSCE, *n* = 4), and Combustion-CHN (SU, *n* = 4; URI, *n* = 10). (The symbol *n* represents the respective number of measurements for each mean.)

c H_2O Extraction, Combustion-Manometry (NIST).

d Information value. For a single method, with just one degree of freedom (*n* = 2) and no additional uncertainty information, NIST has chosen to tabulate only the median (mean), as an information value. This is justified because of the extreme variation of the expanded uncertainty (95 % confidence interval) in this case, i.e., greater than a factor of 30 (90 % interval) for normal data, and the minimal impact of the Central Limit Theorem. The alternative, which NIST does not recommend, despite its statistical validity for normal, random data, is to use a coverage factor of 12.7 to construct the (95 %) expanded uncertainty.

e H_3PO_4 Acidification Manometry (NIST).

f Reference value, which equals the mean value of *n* measurements (*n* is number in parentheses). Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty.

g Soxhlet Extraction (EPA). Although the mass fraction of extractable carbon in dichloromethane given here is consistent with that given in Table 11, the compounds extracted in the two cases are not necessarily the same. The numerical similarity may be fortuitous because different solvents were used for the extractions.

h Information values for the fraction of "elemental" carbon (EC/TC) are derived from an interlaboratory and intermethod comparison [26,54]. Three broad classes of methods were represented: optical (absorbance, reflectance), dry oxidation ("thermal"), and wet oxidation ("chemical"). Also represented were hybrid methods (thermal-chemical, thermal-optical) and kinetic modeling approaches. One conclusion of the exercise is that one should not expect consistent results across methods, because they are (appropriately) measuring different regions of the "black carbon spectrum," which ranges from char at the one extreme to graphitic soot at the other. In this table, NIST presents only the principal features of the results, namely the range observed, and three clusters of EC/TC fractions which together included about 70 % of the data. Exploratory descriptive statistics (medians, interquartile ranges) are used to characterize the three clusters [55]. A complete exposition of the structure and results of the exercise is given elsewhere [26].

Table 13. Values for ^{14}C Isotopic Carbon Composition of SRM 1649a

This table includes reference and information values for ^{14}C speciation in selected chemical components of SRM 1649a. Reference values for ^{14}C in selected chemical fractions are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures. Results for *Elemental and Insoluble Carbon*, which currently do not satisfy the criteria for reference values, are provided as information values.

Isotopic Component	^{14}C, fraction of modern (f_M)[a]		
Total Carbon (TC)[b]			
Combustion-Manometry (NIST)	0.61	±	0.08 (Poisson)
H_3PO_4-Combustion-Manometry (UCI)	0.505	±	0.006 (Poisson)[c]
Combustion-GC (NIST)	0.517	±	0.007 (Poisson)[d]
Elemental Carbon (EC)[e]			
Thermal oxidation/residue (WHOI)	0.065	±	0.014 (*n* = 3)
Chemical oxidation/residue (UCI)	0.153	±	0.004 (Poisson)
Thermal kinetic oxidation/intercept (NIST)	0.038	±	0.024 (Poisson)
Insoluble Carbon[f,g]	0.56		(*n* = 2)
Polar Carbon[b,h]	0.43	±	0.02 (Poisson)
Extractable Carbon[b,i]	0.33	±	0.04 (*n* = 3)
Aliphatic Carbon[b,h]	0.024	±	0.012 (Poisson)
Aromatic Carbon[b,j]	0.17	±	0.08 (Poisson)
Individual PAH[b,h]			
Phenanthrene	0.0406	±	0.0096 (Poisson)
Methylphenanthracenes	0.0434	±	0.0112 (Poisson)
Fluoranthene	0.0637	±	0.0051 (Poisson)
Pyrene	0.0372	±	0.0043 (Poisson)
Benz[*a*]anthracene	0.0413	±	0.0073 (Poisson)
Chrysene/Triphenylene	0.0553	±	0.0059 (Poisson)
Benzofluoranthenes (*b,j,k*)	0.0842	±	0.0053 (Poisson)
Benzo[*ghi*]perylene	0.0864	±	0.0090 (Poisson)

[a] The fraction of modern carbon (f_M) is derived from the ^{14}C/^{12}C ratio observed, relative to 0.95 times that of the contemporary (oxalic acid) standard for radiocarbon dating, SRM 4990B. For detailed information on the definition of f_M, taking into account adjustment for the ^{13}C/^{12}C ratio (for precise work) see [56,57]. Note that all f_M values are corrected for ^{14}C decay to the mid-date of sampling (1977.0) using the physical mean life: 8268 years. Corrections are quite small, with correction factors ranging from 1.00073 to 1.00273. Note that f_M reflects the ^{14}C content compared to the artifactually (SRM) defined reference state "Modern." At the time of collection of SRM 1649a (1976-1977) the ^{14}C content of the living biosphere was approximately 1.35 x Modern as a result of atmospheric nuclear testing [26].

[b] Reference value, which equals the mean value of *n* measurements (*n* is number in parentheses). Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [51], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. For those ^{14}C reference values based strictly on "counting statistics," (*n* =) is replaced by (*Poisson*), and the expanded uncertainty is taken as 1.96 times the Poisson standard uncertainty. This approach was not taken if replication showed a significant non-Poisson component of variance. (Note that AMS ^{14}C uncertainties lacking external replication [independent samples] are treated as Poisson uncertainties for the purpose of this certificate [58].)

[c] The corresponding d ^{13}C value is -25.5 ± 0.2.

[d] The corresponding d ^{13}C value is -25.18 ± 0.12.

[e] Information values. Although the ^{14}C measurements, *per se*, might qualify as reference values having the stated expanded uncertainties, values for ^{14}C speciation in EC must be considered as information values because of their dependence on the fraction of residual carbon (RC), and lack of consensus on the quantitative link between RC and EC. For the Thermal oxidation/residue method, a weighted mean is given, based on relative weights from the individual values and their expanded *Poisson* uncertainties: 0.0606 ± 0.0027, 0.0793 ± 0.0074, and 0.0663 ± 0.0024.

[f] Information value. For a single method, with just one degree of freedom (*n* = 2) and no additional uncertainty information, NIST has chosen to tabulate only the median (mean), as an information value. This is justified because of the extreme variation of the expanded uncertainty (95 % confidence interval) in this case, i.e., greater than a factor of 30 (90 % interval) for normal data, and the minimal impact of the Central Limit Theorem. The alternative, which NIST does not recommend, despite its statistical validity for normal, random data, is to use a coverage factor of 12.7 to construct the (95 %) expanded uncertainty.

[g] Information value, as is the Insoluble Carbon mass fraction. The individual ^{14}C (f_M) values and Poisson expanded uncertainties are 0.571 ± 0.008 and 0.543 ± 0.008 [29].

[h] Soxhlet extraction/LC/PCGC (WHOI, NIST).

[i] Soxhlet Extraction (EPA).

[j] Soxhlet Extraction/LC (NIST). Fraction isolated by normal-phase LC representing the fraction in which the majority of the PAHs elute. (See the Preparation and Analysis section.)

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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

APPENDIX A

The laboratories and analysts listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1649a (Table 7).

Alta Analytical Laboratory, Inc., El Dorado Hills, CA, USA: W.J. Luksemburg
Axys Analytical Services Ltd., Sidney, British Columbia, Canada: L. Phillips
Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada: G. Reuel
Environment Québec, Laval, Québec, Canada: C. Brochu
Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada: G. Poole
GSF National Research Center for Environment and Health, Neuherberg, Germany: B. Henkelmann
Institute of Environmental Chemistry, Umeå University, Umeå, Sweden: R. Anderson
Maxxam Analytics, Inc., Mississauga, Ontario, Canada: C. Lastoria
Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada: E. Reiner
Research and Productivity Council, Fredericton, New Brunswick, Canada: J. Macaulay
Texas A&M University, College Station, TX, USA: T.L. Wade
Wellington Laboratories, Guelph, Ontario, Canada: C. Tashiro
Wright State University, Dayton, OH, USA: T.O. Tiernan

The laboratories and analysts listed below contributed to the results presented for particulate and isotopic carbon composition for SRM 1649a (Tables 12 and 13).

Atmospheric Aerosol Research, Lawrence Berkeley National Laboratory (LBNL), Berkeley, CA, USA: T. Novakov and T. Kirchstetter

Centre des Faibles Radioactivites (LSCE), CNRS-CEA, Gif sur Yvette, France: H. Cachier

Department of Earth System Science, University of California (UCI), Irvine, CA, USA: C.A. Masiello and E.R.M. Druffel

Department of Marine Chemistry and Geochemistry, Woods Hole Oceanographic Institution (WHOI), Woods Hole, MA, USA: C.M. Reddy, T.I. Eglinton, and A. Pearson

Energy and Environmental Engineering Center, Desert Research Institute (DRI), Reno, NV, USA: S.D. Kohl and J.C. Chow

Environmental Research Institute, Stockholm University (SU), Stockholm, Sweden: O. Gustafsson

Graduate School of Oceanography, University of Rhode Island (URI), Narragansett, RI, USA: P.C. Hartmann and J.G. Quinn

Institute for Analytical Chemistry, Vienna University of Technology (TUW), Vienna, Austria: H. Puxbaum and H. Schmid

National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA: B.A. Benner, Jr., L.A. Currie, J.D. Kessler, D.B. Klinedinst, G.A. Klouda, J.V. Marolf, A.E. Sheffield, J.F. Slater, and S.A. Wise

School of Oceanography, University of Washington (UW), Seattle, WA: K.M Prentice and J.I. Hedges

Sunset Laboratories, Inc. (SLI), Forest Grove, OR, USA: R. Cary

U.S. Environmental Protection Agency (EPA), National Exposure Laboratory, Research Triangle Park, NC, USA: R.B. Zweidinger

Accelerator measurements were performed at the National Science Foundation Accelerator Mass Spectrometry (AMS) Facility, University of Arizona (UA), Tucson, AZ; Lawrence Livermore National Laboratory (LLNL), Livermore, CA and the National Ocean Sciences AMS Facility (NOSAMS), Woods Hole Oceanographic Institution (WHOI), Woods Hole, MA.